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QUANTITATIVE STUDIES BY OPTICAL SPECTROSCOPY OF ENERGY EXCHANGE MECHANISMS IN SIMPLE GASES AND SOLIDS

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Abstract

Work has continued in three general directions: condensed phase experiments, emission studies of SiN and N_2^+, and plasma analysis.

Several results have now been obtained from the condensed phase studies. Improvements in apparatus have allowed measurement of free carrier mobilities in solid and liquid argon and krypton. Effects of impurities and temperature variation on these mobilities have been studied. As a preliminary to the study of scattering processes, using laser beams, in liquids and van der Waal's solids, equipment and techniques have been developed, which have allowed observation of Raman and Brillouin scattering in CS_2 and benzene liquids. Charge transfer processes in the benzene-iodine complex are being studied at low temperatures using absorption methods. Initial results reveal problems in analysis due to scattering at the complex film surface.

The study has continued of flowing gas systems where the initiating energy source is an electrical discharge. The addition of volatile silicon compounds to active nitrogen has yielded new information on the electronic states of SiN, including the existence of a large vibration population inversion in the $B^2\Sigma$ state of that compound. In a study of N_2^+, a detailed perturbation calculation on the mutual interaction between the v = 1 level of the $B^2\Sigma$ state and the v= 11 level of the $A^2\Pi$ state has been completed. Order of magnitude calculations for the interaction predict energy shifts comparable with experimental values. Populations of vibrational and electronic states of N_2^+ excited in a helium afterglow have been obtained over a large pressure range.

A spectroscopic and Langmuir probe study of the hydrogen negative glow plasma has been completed. Electron density and temperature measurements indicate that the plasma is beam generated and diffusion controlled. A value for the ambipolar diffusion coefficient has been deduced.

FREE CARRIER MOBILITY STUDIES IN SOLID RARE GASES

H. D. Pruett

Abstract

The free carrier mobility has been measured in argon and krypton in solid and liquid phases. A saturated electron drift velocity similar to the "hot electron" phenomenon in semiconductors has been observed in both of these rare gas solids. Temperature dependence and effect of impurities on electron mobility have been measured.

Objective

The objective of these investigations is to determine the nature of a few simple free-carrier interactions in rare gas liquids and solids.

Program

1. Measure the field and temperature dependence of the free carrier drift velocity in neon and xenon in both liquid and solid phases.
2. Compare results in neon and xenon with those obtained in argon and krypton.
3. Continue work on theoretical model to explain the results obtained with argon and krypton.

Summary of Progress

The apparatus which was used for these investigations is shown in Figure 1. . The lower section of the rack contains the high vacuum system which was used to evacuate the electrode cell to at least 10^{-6} Torr. At the top of the rack is the 1-10 KV power supply. On the left are the two mercury manometer - bubblers; one was used to measure the vapor pressure of the sample, the other was used to measure the vapor pressure of the refrigerant. In the center, the glass section hanging down from the dewar cap is the electrode cell. The dewar (not shown in this photograph) which holds the refrigerant has

two vertical unsilvered slits so that visual observation of the crystal growth process can be made.

At the beginning of this period, problems which had prevented quantitative measurements were solved. By careful electrical shielding, troublesome electrical transients from fluorescent lighting were reduced to a tolerable level. A time-averaging technique to improve the signal-to-noise (S/N) ratio was devised. The technique utilizes the intensity integrating properties of photographic film in conjunction with signal pulses displayed on an oscilloscope screen.

Because of the low level ($\geq 25 \times 10^{-6}$ volts) and fast risetime (100×10^{-9} sec) of the signal pulse, it was necessary to design and build a low noise, wideband, voltage-sensitive preamplifier. The preamplifier which was developed has an equivalent noise input resistance of 200 ohms and a risetime of 10 nanoseconds. The preamplifier amplifier-oscilloscope combination which was constructed has a maximum sensitivity of 5 microvolts/cm; however the preamplifier noise level prevents the use of the maximum sensitivity.

The drift velocity of electrons has been measured in argon and krypton in both solid and liquid phases. The temperature dependence of the drift velocity has been measured in both of these materials. Because of the expense of krypton, effects of impurities on the electron drift velocity were measured only in argon.

In the course of the measurements, crystal growth techniques were evaluated in terms of reproducibility of results. The relationship between crystal growth rate and high electric field breakdown was examined. The breakdown phenomena have been qualitatively explained in terms of thermal instability[1].

In order to investigate the possibility of purifying rare gas solids by zone refining, experimental apparatus was constructed and preliminary studies have been initiated. Evidence that krypton can be zone refined was obtained during the drift velocity measurements; however, the zone refining experiments were restricted to argon due to cost considerations.

From experience gained during studies in argon and krypton, a new electrode assembly was constructed for use with xenon and neon. The principal modification was to reduce the size of the electrode cell in order to minimize the quantity of rare gas required for an experiment. In the new cell, only about one liter of rare gas is required for each experiment.

A theoretical investigation of the interaction mechanism which is responsible for the saturated electron drift velocity in argon and krypton is in progress.

In semiconductors, notably germanium and indium-antimonide, the electron drift velocity has been observed to saturate in high electric fields. The theoretical explanation[2] for these materials is only partly applicable to the rare gas solids; therefore additional attention must be given this problem.

An abstract has been submitted to the American Physical Society for a talk to be given at the September meeting of the society.

Future Plans

During the month of June, the electron drift mobility measurements will be completed in xenon and neon. It is hoped that the results in these two materials will be useful in evaluating the model which is currently being developed to explain the saturated electron drift velocity in argon and krypton.

The results obtained in June will be incorporated into a dissertation which is being prepared for submission in partial fulfillment of the requirements for the Ph.D. degree at the University of California, Santa Barbara.

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SCATTERING OF LASER LIGHT FROM PHONONS

S. L. Shapiro

Abstract

Recently, at the National Bureau of Standards, Boulder, Colorado, Dr. D. A. Jennings and I, a guest worker, were able to observe normal Brillouin scattering in special high scattering materials. Studies of Brillouin and Raman scattering in other materials will be undertaken this summer to determine the best systems to be used for further measurement.

Objective

The objective of these investigations is to explore the interaction of laser light with phonons in the visible and acoustic region in liquids and in van der Waals crystals.

Program

1. Build equipment and develop techniques for observing Raman and Brillouin scattering.
2. Seek for ordinary Brillouin scattering in solid argon in order to accurately determine the longitudinal and transverse velocities of sound and perhaps the damping of the phonons in the crystal.
3. Look for the rotational Raman effect of methane in krypton and also oxygen in krypton and argon.
4. Look for the inverse Raman effect in liquid oxygen.
5. Possibly look for rotational Raman effect due to rotating complexes in liquid helium-3 below the λ point.

Summary of Progress

In the last six months we have built equipment and developed techniques for observing scattering processes. Using a 10 milliwatt laser at 6328A we were able to duplicate the results of Chiao and Stoicheff. Brillouin scattering has been observed in CS_2 and benzene liquids. A Fabry-Perot interferometer was used to separate the shifted Brillouin light

from the unshifted laser line. With properly chosen polarization and Fabry-Perot mirrors the patterns were easily visible to the naked eye.

We initially had difficulty in observing the Brillouin scattered light because we chose the wrong polarization of the laser beam. The beam polarization must be chosen so that the electric field vector is parallel to the Fabry-Perot. The whole medium oscillates like a gigantic dipole and hence there is no radiation along the electric field vector. One must also be careful to select mirrors for the Fabry-Perot of high transmission and high finesse. Mirrors with 85 to 90% reflectivity have been found to work well.

A high intensity argon laser is being built in Boulder and will be used to study Brillouin and Raman scattering. The laser has been operated in the pulsed mode and recently c.w. at low power. In order to get the laser to go c.w. a long helical tube was connected between the anode and the cathode. Without this tube ions were pumped to the electrodes and a differential pressure was built up. A magnetic field must be used to confine the discharge to the center of the quartz tube at higher output powers in order to extend the life of the tube. The magnetic field also will increase the power output significantly. Should we succeed in operating the argon laser at high enough intensities we may use it to look for Brillouin scattering in argon, rotational Raman scattering of methane in krypton, and inverse Raman scattering by oxygen.

The He-Ne laser now operating at 10 to 15 mW will be filled with helium 3 for higher gain. Also a new and longer He-Ne tube has been designed and may be available for similar scattering studies.

Using a Q-switched ruby laser (about 10 Megawatts) stimulated Brillouin scattering has been observed in certain liquids. An attempt to observe stimulated Brillouin scattering in crystalline argon ($77^{\circ}K$) and in liquid argon failed, indicating a higher threshold for stimulated Brillouin scattering in these materials. Similar attempts in liquid N_2 also failed. In the course of these studies the quartz windows of the cell were pierced with holes as if drilled, but with some cracking.

A lower threshold was expected because phonon losses were expected

to be cut down at 77°K as compared to room temperature materials. However, a recent theory by Kroll suggest that other factors play a role.

Future Plans

Continue work on understanding interactions between phonons and photons and perhaps do some work on phonon-electron scattering.

CHARGE TRANSFER PROCESSES IN ELECTRON DONOR-ACCEPTOR COMPLEXES IN SOLIDS AT LOW TEMPERATURE

J. S. Margolis

Abstract

An investigation into the formation of electron donor-acceptor complexes is being performed. The technique is novel in that the complexes are formed and their spectra taken without the perturbing effects of usual solvents. The vapors of the constituents of the complex are allowed to condense on a cold window and form a thin film through which absorption spectra may be taken. Data so far obtained indicate that the complex may be formed in sufficient concentrations to observe the spectra.

Objective

The charge transfer spectra of electron donor-acceptor complexes are quite intense and in order to do satisfactory spectroscopy on them it is necessary to make thin films of the complex or to observe them in a dilute solution of some spectroscopically inert solvent. The various kinds of inert solvents used for this purpose show differing effects on the spectra indicating varying degrees and kinds of inertness. A simpler system to study, presumably, would be the complex unperturbed by foreign species and it is possible to obtain this by making very thin samples of the pure material. This is done in the present study by condensing the electron donor-acceptor complex onto a cold window. In this way two ends are accomplished: the first in that the spectra of the pure material is obtained and the second is that it is possible in this way to study the formation of the complex. This latter is done by depositing the constituents separately on the cold window; this then hopefully allows an insight into how the acceptor molecule is trapped in the donor matrix and forms the complex.

Program

The program anticipated at this time is to study temperature effects on the spectra by going to lower temperatures - using liquid hydrogen

or helium to cool the windows. It must also be seen whether annealing, or forming the films at a rather high temperature and then cooling them will have an effect on the spectra. It is anticipated that using different acceptors will bring out important information and some of these are on hand to try.

Summary of Progress

Spectra have been taken of the benzene iodine complex which was made in two different ways and both of which give similar results. The complex was made by dissolving iodine in benzene and allowing the complex formed in this way to evaporate into the vacuum space of a dewar. Also the complex was formed by allowing benzene and iodine separately to evaporate into the dewar and condense onto the windows. This was done at 77°K indicating that it takes very little energy for the formation of the benzene iodine complex.

Future Plans

The spectra so far obtained are incomplete in that they do not allow a determination of the shape of the charge transfer absorption band. The short wavelength side of the absorption is obscured, presumably because of the large amount of scattering of the UV light at the surface of the film. Some way will have to be found to improve the films so that the shape of the absorption may be determined. Several kinds of acceptors, such as Br and Cl will be tried.

CHEMILUMINESCENT EMISSION FROM THE REACTIONS OF VOLATILE SILICON COMPOUNDS WITH ACTIVE NITROGEN

K. Schofield

Abstract

A spectroscopic investigation has been made of the chemiluminescent emission from atomic flames produced by the addition of volatile silicon compounds to active nitrogen. Of seven such additives, $SiCl_4$ proved to be the most intense source for SiN emission. The two electronic systems of SiN, the $B^2\Sigma - X^2\Sigma$ and $C - A^2\Pi$, were detected both in the reaction vessel glow and also in an intense glow from a liquid nitrogen trap.

An analysis of the relative vibration populations in the $B^2\Sigma$ electronic state of SiN showed large population inversions with a maximum population in the $v = 5$ level. A strong rotational perturbation of the $B^2\Sigma - X^2\Sigma$ SiN system at $v' = 4$ in the $K' = 13$, 14, 19 and 21 levels indicated a leakage occurring from these perturbed levels into those of the perturbing state.

The finding that bands of the $C - A^2\Pi$ system of SiN in the trap glow were enhanced in intensity relative to the $B^2\Sigma - X^2\Sigma$ system has made a reinvestigation of the system possible. New bands of this system are reported and analysis shows the need to increase the original numbering of v" by one.

Two new and unidentified spectra have been recorded which result from the addition of silicon compounds to active nitrogen - a green system in the 5180 to 6020A region, produced by $SiCl_4$, and an orange chemiluminescence, 5540 to 7250A, through the addition of $SiBr_4$.

Objective

To gain insight into the nature of the collision processes and energy exchange mechanisms operative in the gaseous phase.

Program

1. Analyse the new information on the $B^2\Sigma - X^2\Sigma$ SiN electronic system.

This concerns data on the rotational and vibrational population distributions of the $B^2\Sigma$ state.

2. Check the correctness of Mulliken's[1] vibrational analysis of the C - $A^2\Pi$ transition of SiN.

3. Attempt analyses of the two new systems that have been observed. The green system lies from 5180 to 6020A and the orange system from 5540 to 7250A.

Summary of Progress

The chemiluminescent emission analysed in this research has been produced by an atomic flame technique. A picture of the apparatus used is shown, Figure 2. A small microwave cavity produces the active nitrogen which then passes into the reaction vessel. A volatile silicon compound is drawn into the system through a needle valve and forms an atomic flame or afterglow in the reaction vessel. Observations can be made through the quartz window on the end of this. Liquid nitrogen traps placed in the line to collect the silicon material have also played a part in this research since under certain conditions strong emission could also be produced from them.

The results obtained with this equipment were recently presented at the "Symposium on Chemiluminescence"[2] and have now been accepted for publication in a special edition of "Photochemistry and Photobiology" which is covering the symposium.

Future Plans

1. A further investigation of the Mulliken bands of SiN is planned. The enhancement of emission of these bands in a low temperature trap source is to be examined for a temperature dependence. Traps of different design are being investigated to see if any improvement can be made in light gathering efficiency so that studies under higher resolution can be made.

2. A strong turquoise colored afterglow can be obtained by adding traces of $GeCl_4$ to active nitrogen. By analogy with CN and SiN this appears to be the $B^2\Sigma$ - $X^2\Sigma$ electronic transition of GeN. The spectrum

of a natural mixture of GeN is too complicated due to the isotope problem but an attempt to purchase isotopically pure $GeCl_4$ is at present underway.

3. During this research, under certain conditions, while using $SiCl_4$ and $GeCl_4$ additives, 'catalysed' emission of N_2 has been observed. In both cases the emission arose when addition was stopped. This consisted of a strong orange glow filling the whole reaction vessel. Pressures around 2 mm Hg were necessary. The glow gradually dies away but lasts as long as ten minutes. With $SiCl_4$, the glow consists of 1st positive bands of nitrogen. However, the intensity distribution of these results from a more even distribution over the $v' = 6$ to 12 levels, so changing the emitted color from that usually associated with the Lewis-Rayleigh afterglow.

With $GeCl_4$, 1st positive bands of nitrogen with a similar intensity distribution were again observed but in this case 2nd positive bands of nitrogen and the 1st negative system of N_2^+ were also apparent.

Further study of this effect may lead to new information on the collision mechanisms that can occur in active nitrogen.

Unfortunately my time on this project is coming to an end but it is hoped that my replacement may pursue these lines of research. At present Dr. Broida and myself are concluding a review type article entitled "Flame Kinetic Studies", to be published next year by Academic Press, Inc., N. Y., in their series "Methods of Experimental Physics". It will appear in Volume VIII "Atomic Physics". The basic concept of the article is to provide an up to date source of reference on flame kinetic studies, covering all the important methods and techniques. Limitations of applicability and accuracy are discussed.

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POPULATION INVERSION IN THE $B^2\Sigma$ STATE OF N_2^+

J. L. Dunn

Abstract

A detailed perturbation calculation on the mutual interaction between the v = 1 level of the $B^2\Sigma$ state and the v = 11 level of the $A^2\Pi$ state is complete. Order of magnitude calculation for the interactions centered about K = 2 and 13 predict energy shifts comparable with experiment. Relative intensity measurements of the rotational, vibrational, and electronic transitions of nitrogen excited in a helium afterglow have been made for a pressure range of 0.07 to 24 mm Hg and helium flow rates of 0.4 to 180 cm^3/sec.

Objective

Quantitative measurements of separations and relative intensities of the perturbed levels in the $B^2\Sigma$ state of N_2^+ and methods for producing population inversions in these levels are desired. With the relative intensity data, it should be possible to formulate a set of rate equations from a simple kinetic model which describe how the perturbed energy levels are populated or depleted by radiation, collisional, and perturbation processes. A feasibility study of possible microwave double resonance experiments can then be undertaken.

Program

1. Measure relative intensities of the perturbed, unperturbed and extra rotational components in the (1,n) and (3,n) bands of the $B^2\Sigma - X^2\Sigma$ transition as a function of pressure, helium flow rate, and microwave power applied to the helium discharge and afterglow region.
2. Measure relative intensities of the atomic and molecular emission produced when nitrogen is added to a helium afterglow.
3. With the data from the relative intensities as well as the rotational perturbation information, choose a kinetic model from which the desired rate equations can be written down.

4. If feasible, attempt to obtain microwave spectra among the perturbed and neighboring unperturbed levels by the microwave - optical technique (double resonance) to determine the hyperfine structure and the dipole moment of the electronically excited states.

Summary of Progress

Figure 3 shows the flow system used in the experiment. Ultra pure helium flows through the activated charcoal coil trap which is maintained at liquid nitrogen temperature. The gas then expands in a nozzle where a discharge is produced with a 2450 Mc/sec source feeding a 1/4 wave air-cooled cavity. Prepurified nitrogen is added to the helium afterglow via the needle valves at the region of the quartz window. Downstream, the pressure is measured with a MKS Baratron differential type pressure meter.

The detailed perturbation calculation on the mutual interaction of the nearly degenerate v = 11 level of the $A^2\Pi$ state and the v = 1 level of the $B^2\Sigma$ state is complete. Although measurements of the multiplet splitting due to the rotational perturbation have been made in this laboratory, the data of Coster and Brons[1], Crawford and Tsai[2], and Wood and Dieke[3] will be used, since their high resolution measurements appear to be quite satisfactory for the calculations of the microwave transition probabilities.

Attempts to observe a "perturbed" band of the $A^2\Pi - X^2\Sigma$ transition with v' = 11 was negative. Although the calculated wavelengths for the expected strong bands with v' = 11 were located in wavelength regions of strong N_2^+ $(B^2\Sigma - X^2\Sigma)$ emission, both photoelectric and photographic techniques failed to produce any desired bands.

A systematic study of the relative intensity distribution of atomic and molecular nitrogen and helium emission spectra, when nitrogen is added to a helium afterglow, has been undertaken in the region 2000 to 8600A. Strong emissions were observed from the N_2^+ $(B^2\Sigma - X^2\Sigma)$ bands as well as strong bands corresponding to the $A^2\Pi - X^2\Sigma$ transition in N_2^+. The first positive bands of N_2 were also observed while atomic

nitrogen emission was observed for a particular set of experimental variables. Electronic, vibrational, and rotational intensity distributions for the various transitions have been undertaken as a function of the experimental variables. The main experimental variables are pressure, helium flow rate, relative concentrations of added nitrogen, and microwave power applied to the helium discharge. A double-pass 0.75 meter Fastie-Ebert monochromator with wide slits (500 micron) is being used. The relative response of the monochromator (grating, photomultiplier, etc) as a function of wavelength was determined using a tungsten source of known temperature and emissivity.

Future Plans

The relative intensity measurements will be continued to cover the following range of variables: pressure, 0.07 to 40 mm Hg; helium flow rate 0.4 to 400 cm^3/sec NTP; microwave power applied to helium discharge, 5 - 60 watts; and relative nitrogen concentrations (e.g. nitrogen flow rates) 0.2 - 2.0 cm^3/sec NTP.

From the relative intensity and rotational perturbation data, develop a kinetic model from which the desired rate equations can be deduced.

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THE NEGATIVE GLOW PLASMA

G. A. Woolsey

Abstract

A spectroscopic and Langmuir probe study of the negative glow plasma in hydrogen has been completed. Some preliminary observations of the deuterium plasma have been made.

Objective

To obtain information on the mechanism and properties of the negative glow plasma.

Program

1. Form a negative glow plasma in hydrogen with as large a charge-carrier density as possible.
2. Examine the plasma spectrum under a variety of experimental conditions.
3. From probe measurements obtain information on electron and ion densities and energies, and on the collision processes taking place in the plasma.
4. Investigate the effect on the plasma of an axial magnetic field.
5. Compare the plasma formed at liquid nitrogen temperature with that at room temperature.
6. Compare the hydrogen negative glow plasma with those of other gases such as helium and deuterium.

Summary of Progress

The work on hydrogen has been completed and is reported in a paper submitted to the Journal of Applied Physics. Comparison with the helium plasma is made in this paper.

Figure 4 shows the quartz discharge tube used for spectroscopic analysis, together with the palladium leak for purifying hydrogen or deuterium. Figure 5 is a general view of the apparatus used in the probe study of the plasma.

Initial observation of the deuterium plasma has shown it to be similar to the hydrogen glow both visually and spectroscopically. A new problem encountered is the slow rate of leak of deuterium through heated palladium. This necessitates a much slower flow rate than was possible with hydrogen, causing greater contamination problems.

Future Plans

In the study of the deuterium plasma, an important measurement will be that of the ambipolar diffusion coefficient. A comparison of the coefficients for hydrogen and deuterium will yield information on the mass effect.

Future plasma studies in oxygen and nitrogen are planned.

An attempt will be made to form carbon vapor in an inert atmosphere of the order of 1 mm Hg using the helium negative glow plasma. By confining with a magnetic field the high energy electron beam which enters the plasma from the cathode dark space, and allowing it to bombard a sample of carbon, it may be possible to obtain high enough temperatures for vaporization. Carbon vapor may then be obtained in helium at pressures near 1 mm Hg. By using this source of carbon vapor in a flow system, other vapors and gases may be added downstream, and a spectroscopic study made of the resultant chemical reactions.

FIGURE CAPTIONS

1. Photograph of apparatus used for free carrier mobility studies in the rare-gas-solids.

2. Photograph of atomic flame apparatus used for measurement of SiN.

3. Flow system apparatus for studying nitrogen excited in a helium afterglow.

4. Quartz discharge tube used for spectroscopic analysis of discharge plasma.

5. General view of the apparatus used for probe analysis of the plasma.

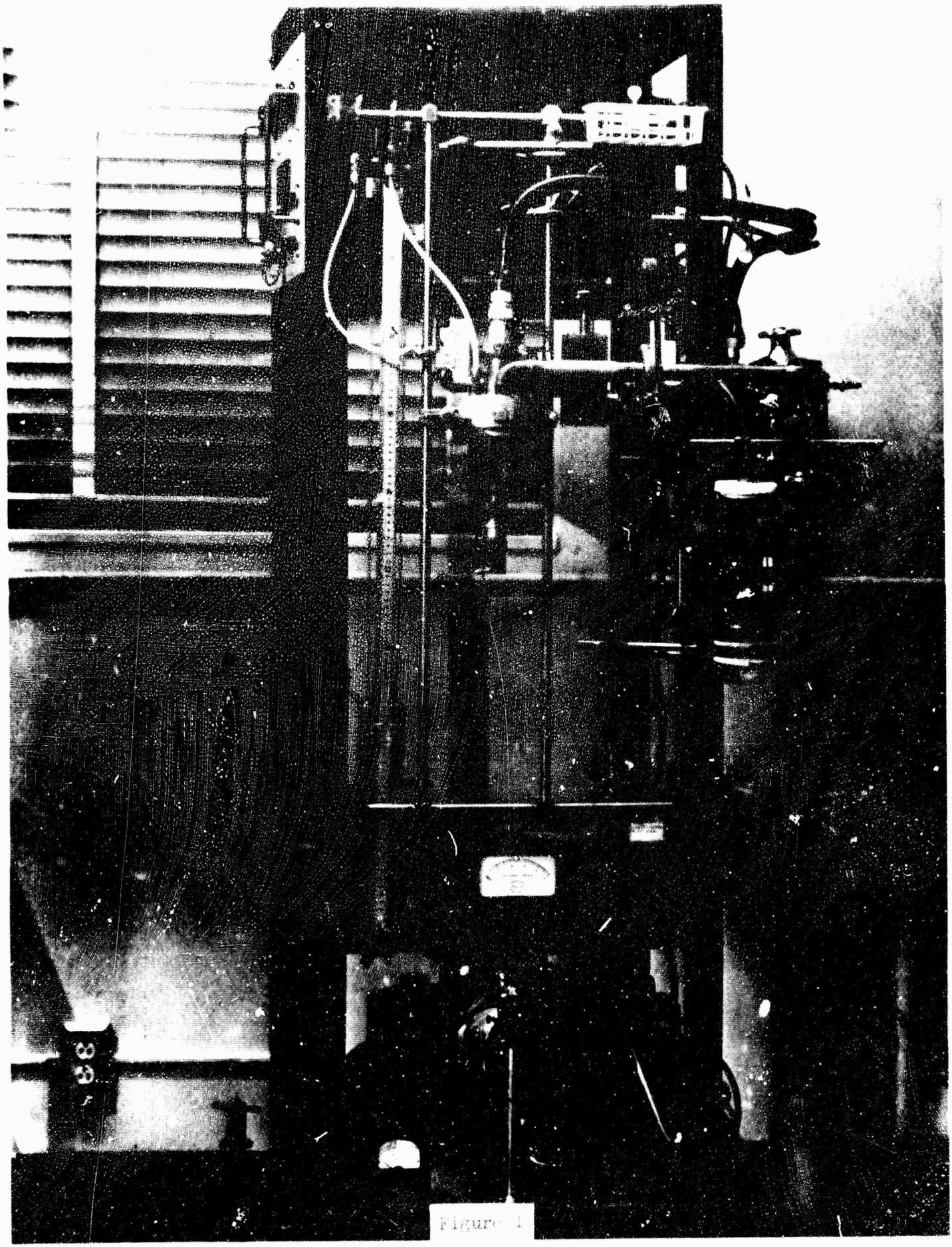
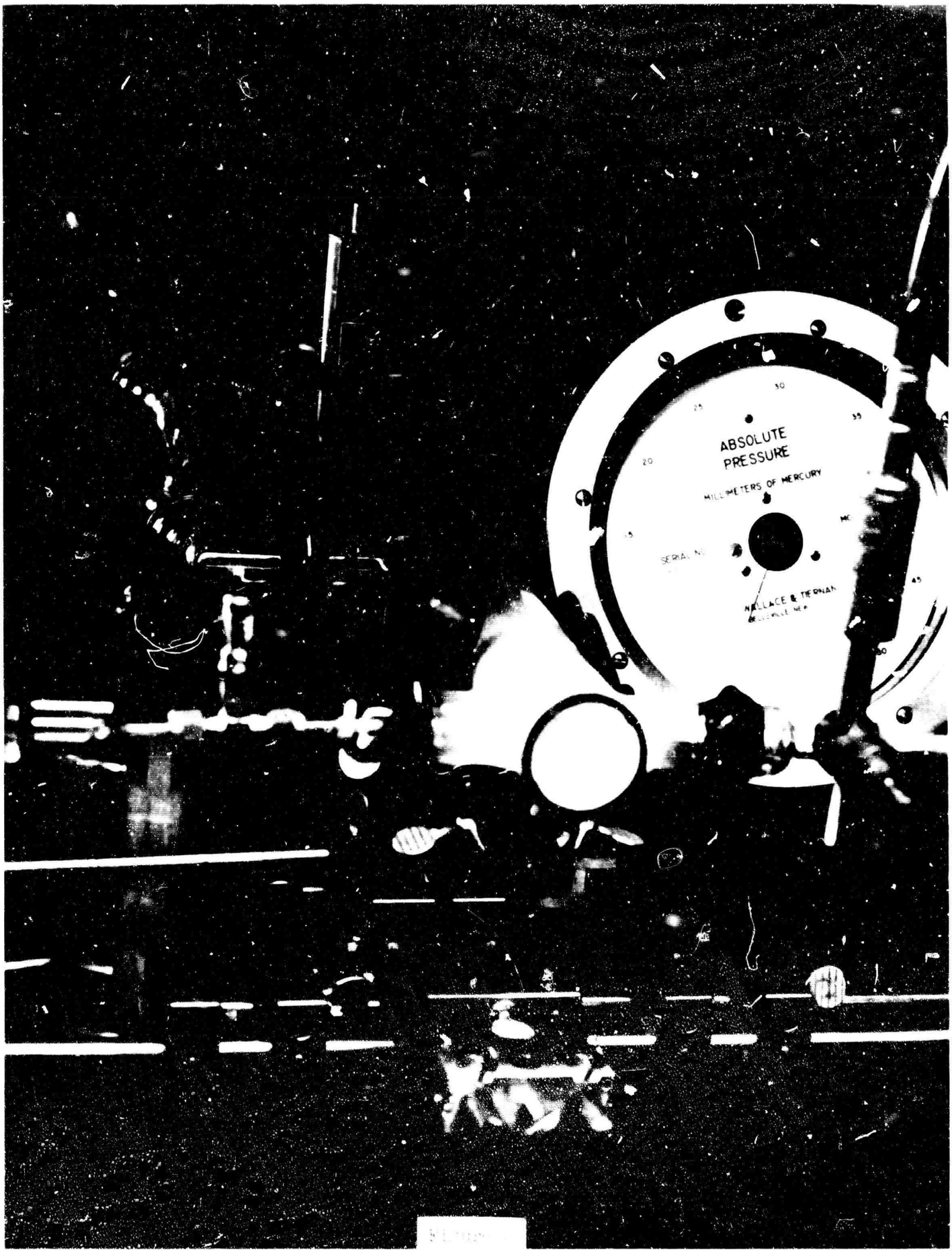


Figure 1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MILLIMETERS OF MERCURY

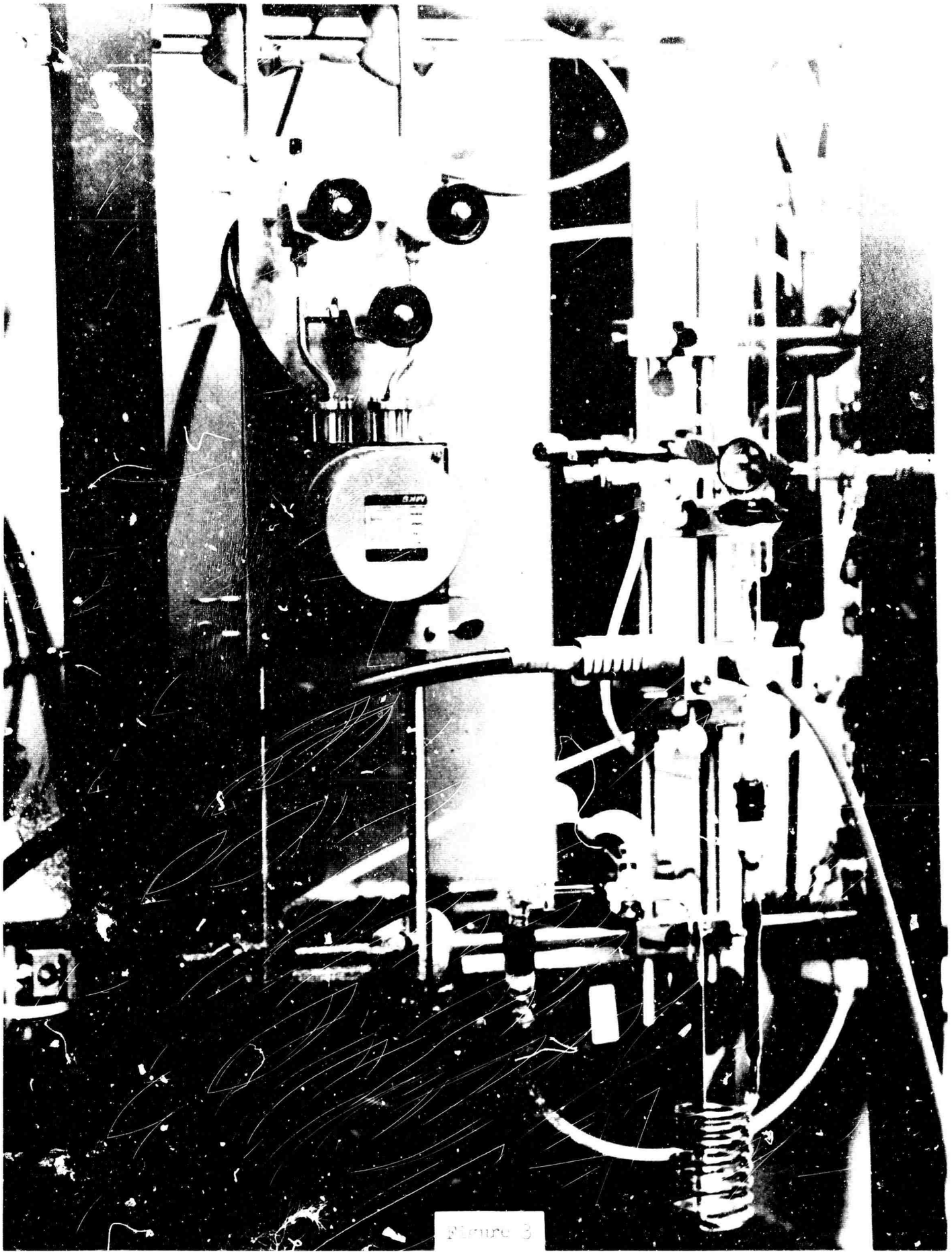

Figure 3

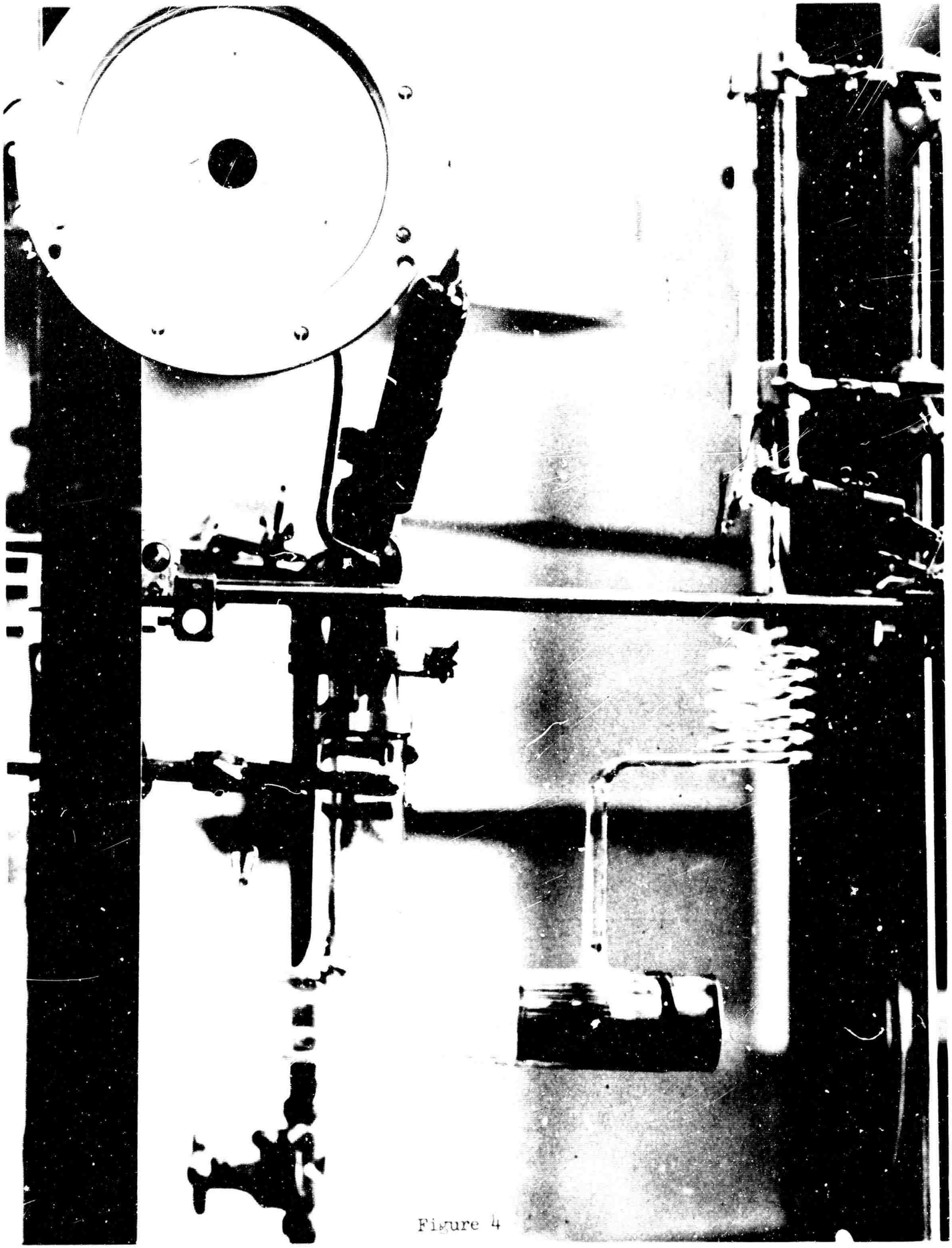

Figure 4

Figure 3

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